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APPENDIX J

SHORT CHECKLISTS FOR ON-SITE LABORATORY INSPECTIONS

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SHORT CHECKLISTS FOR ON-SITE LABORATORY INSPECTIONS

CHARTS J-1 through **J-10** contains a short version of laboratory inspection checklists. The short checklists are developed to reduce the amount of paper work to be brought to an on-site laboratory inspection. Only the major areas to be examined are listed in the short checklists. They serve as a reminder for an experienced inspector to check the adequacy of laboratory facility, equipment, operation, and QA/QC policy and practice during an on-site inspection. Depending on a laboratory's performance and an inspector's preference, the inspector may choose the long checklists (Appendix I), the short checklists, or a hybrid of both to perform an on-site laboratory inspection.

Short Laboratory Inspection Checklists

CHART J-1 Lab Organization, Personnel, and Management

1. Organization
 - a. Organization chart
 - b. Management structure
 - c. Principal officers
 - (1) Lab Director - ten years
 - (2) Lab Manager - seven years
 - (3) Organic Lab Manager - five years
 - (4) Inorganic Lab Manager - five years
 - (5) QA Officer - five years
 - d. Reporting relationships
2. Personnel
 - a. Resumes
 - b. Job descriptions
 - c. Training program
 - (1) Initial training and evaluation
 - (2) Continuing training and auditing
 - (3) Documentation
 - d. Minimum Experience without supervision
 - (1) GC supervisor - three years
 - (2) GC analysis - one year
 - (3) Pesticide residue analysis - two years
 - (4) GC/MS supervisor - three years
 - (5) GC/MS analysis - one year
 - (6) GC/MS spectral interpretation - two years
 - (7) HPLC analysis (explosives) - one year
 - (8) Organic sample preparation - one year
 - (9) AA/ICP supervisor - three years
 - (10) AA/ICP analysis - one year
 - (11) Metal sample preparation - six months
 - (12) Wet chemistry supervisor - three years
 - (13) UV/VIS analysis (cyanide) - one year
 - (14) IR analysis (TRPH) - one year
 - (15) IC analysis (common anions) - one year
 - (16) Classical analysis - one year
 - (17) Radiochemistry supervisor - five years
 - (18) Radionuclides analyst - two years
 - (19) Gross alpha/beta analysis - six months

CHART J-2 Lab Facility, Equipment, and Instrumentation

1. Facility
 - a. Security
 - b. Sample storage
 - c. Chemical storage
 - d. Bench space
 - e. Number of hoods
 - f. Ventilation
 - g. Document archives
2. Equipment
 - a. Reagent water system (Free from interferents at MDL; resistivity $\geq 16\text{M } \Omega$.)
 - b. Conductivity meters (Daily or before-use calibration check; cell constant determined annually.)
 - c. pH meters (Scaled to ≤ 0.1 pH unit; standardized daily at two pH units that bracket the expected pH range and are no more than three to four pH units apart; temperature compensated.)
 - d. Analytical balance (Capable of weighing to 0.1 mg; daily or before-use check with a minimum of one Class S weight in the range to be used and monthly with a series of Class S weights; $\leq 0.1\%$)
 - e. Class S weights (50 mg to 4 kg; calibrated within five years and traceable to NIST.)
 - f. Drying ovens (Temperature checked before and after each usage.)
 - g. Muffle furnace (Temperature verified annually.)
 - h. Hotplates (Capable of temperature control within $\pm 5^\circ\text{C}$.)
 - i. Water bath (Capable of temperature control within $\pm 5^\circ\text{C}$.)
 - j. Refrigerators (Temperature checked twice daily.)
 - k. Thermometers (Mercury type: scaled to $\leq 1^\circ\text{C}$; checked annually against NIST traceable thermometer at two separate temperatures; Dial-type: calibrated quarterly against NIST traceable thermometer.)
 - l. Autopipetors (Daily or before-use check of delivery volume gravimetrically.)
 - m. Volumetric glassware (Class A segregated from others.)
 - n. Glassware cleaning station (Metals, ammonia, phosphorus, volatiles, and semivolatiles.)
 - o. Sonicator (Titanium horn; 475 watts with pulsing capability.)
 - p. TCLP (ZHE)
 - q. LIMS (Audit trail and security.)
 - r. Safety equipment
 - s. Waste disposal

CHART J-2 Lab Facility, Equipment, and instrumentation
(continued)

3. Instrumentation

- a. AA: Metals (7000s)
 - (1) GFAA with Zeeman correction (As, Pb, Sb, Se, Tl)
 - (2) CVAA (Hg)
 - (3) FLAA
- b. ICP: Metals (6010A)
- c. GC:
 - (1) ECD (8080, 8150A)
 - (2) ELCD (8010A, 8140)
 - (3) FID (8015A, 8040A, 8100)
 - (4) PID (8020)
- d. GC/MS:
 - (1) VOA (8240A, 8260)
 - (2) BNA (8250, 8270A)
- e. HPLC:
 - (1) PAH (8310)
 - (2) Explosives (draft 8330)
- f. IC: Common Anions (300s)
- g. IR: TRPH (418.1)
- h. UV/VIS: Cyanide (9010A, 9012)
- i. Autoanalyzers

4. Backup Instrumentation and Preventive Maintenance

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CHART J-3 Sample Receipt, Storage, and Preservation

1. SOPs
2. Sample Receipt
 - a. Cooler receipt checklist
 - b. Ventilation hood
 - c. External chain-of-custody
 - d. Internal chain-of-custody
 - e. Unambiguous sample number
 - f. Documentation of problems and resolutions
 - g. Coordination with the primary contractor and the USACE
3. Sample Storage
 - a. Temperature controlled ($4\pm 2^{\circ}\text{C}$; thermometer in liquid.)
 - b. Security (Locked storage.)
 - c. Segregation for volatiles and standards
4. Sample Preservation
 - a. Cold storage
 - b. pH preservations (Check and adjust.)
 - (1) $\text{pH} < 2$: Ammonia, COD, hardness, Kjeldahl and organic nitrogen, metals, nitrate-nitrite, oil & grease, organic carbon, total phosphorus, TOX, radiological tests, gross alpha and beta, and total radium.
 - (2) $\text{pH} < 4$: Phenolics.
 - (3) $\text{pH} > 9$: Sulfide.
 - (4) $\text{pH} \geq 12$: Cyanide.
5. Scheduling and Tracking (Sample holding times and client requested suspense dates.)

CHART J-4 Sample Preparation

1. SOPs
2. Chemicals and Reagents:
 - a. Reagent-grade chemicals shall meet the current Committee on Analytical Reagents of the ACS specifications or better and with minimum purity >90%.
 - b. All chemicals and reagents shall be labelled and signed with the date of receipt or preparation.
 - c. All reference materials and measurements shall be traceable to NIST.
 - d. All acids shall be reagent grade or better, except high-purity grade or equivalent for ICP work.
 - e. All solvent shall be chromatographic grade or better.
 - f. All reagent documentation shall indicate:
 - (1) Solvent
 - (2) Concentration
 - (3) Date
 - (4) Preparer's name
 - (5) Expiration date
3. Definition of Batch: Samples of ≤ 20 with similar matrix prepared and analyzed with same technique and reagents at same time or time sequence. Each batch should have a complete set of method required laboratory QC samples.
4. Matrix Types:

a. Surface water	b. Groundwater	c. Wastewater
d. Soil	e. Sediment	f. Sludge
g. Incineration ash	h. TCLP extract	i. Leachate
j. Oil	k. Product	m. Waste
n. Other (Plant, biological, etc.)		
5. Field QC Samples (Blind to analysts.)
 - a. Trip blanks
 - b. Rinsate blanks
 - c. Field duplicates
6. Laboratory QC Samples (5% per batch.)
 - a. Method blanks
 - b. Matrix duplicates
 - c. Matrix spikes
 - d. Matrix spike duplicates
 - e. Lab control samples
 - f. Any other method specific QC samples

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CHART J-5 Sample Analysis

1. SOPs
2. Method Validation
 - a. New method or instrument
 - (1) Accuracy
 - (2) Precision
 - (3) Detection limits
 - (4) Linear calibration ranges
 - b. Modified method or instrument
 - (1) Accuracy
 - (2) Precision
 - (3) Detection limits
 - (4) Linear calibration ranges
3. Instrument Calibration
4. General QA/QC
 - a. System performance audit
 - b. Analyst's performance audit
 - c. Blind QA samples
 - d. Documentation
5. Method Specific Laboratory QC Samples
 - a. Method blanks
 - b. Matrix duplicates
 - c. Matrix spikes
 - d. Matrix spike duplicates
 - e. Laboratory control samples (LCS)
 - f. Other method specific QC samples (ICS, CCS, etc.)
6. Method References: (Promulgated.)
 - a. USEPA SW-846, Revision 0, September 1986:
7000s, 7040/7041, 8020, 8080, 9060.
 - b. USEPA SW-846, Revision 1, July 1992:
6010A, 8010A, 8150A, 8240A, 8270A, 9010A.
 - c. USEPA-600/44-79-020, March 1983:
418.1.

CHART J-5.1 Halogenated Volatile Organic Compounds by GC (8010A)

1. Number of Analytes: 34
2. Preservation/Storage Conditions: Na₂S₂O₅ if chlorine present (aqueous); stored at 4°C.
3. Holding Time: 14 days.
4. Amount for Extraction: 5 mL (aqueous) and 5 grams (solid) by 5030A.
5. Method of Validation:
 - (1) Extract and analyze four replicates of QC check standard. Compare results with Table 3.
 - (2) MDL (Table 1) shall be empirically established and verified semiannually for each matrix.
 - (3) Linear calibration range shall be established and verified semiannually.
6. Standards:
 - (1) Standard Solution Expiration: Stock standards (except gases): six months; stock gas standards: two months; calibration standards: 24 hours if no headspace
 - (2) Internal Standards: Optional; no internal standards specified.
 - (3) Surrogate Standards: Add surrogates (bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane) to encompass range of temperature program. Results within lab established control limits.
 - (4) QC Check Standards: If MS/MSD results fall outside control limits, a QC check standard must be analyzed and fall within those ranges designated in Table 3.
7. Calibration:
 - (1) Initial Calibration: Minimum of five levels with the lowest near but above MDL; linear correlation coefficient ≥ 0.995 . If %RSD < 20, linearity is assumed and average RF may be used.
 - (2) Continuing Calibration: Mid-level calibration standard run every ten samples and at the end of the analytical run. If not within $\pm 15\%$ of predicted response, recalibrate.
8. Analysis: An example of run log is listed below.
 - (1) Initial Batch: (≤ 20 samples of similar matrix.)
 - CCV ($\leq \pm 15\%$)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Table 3 or manufacturer/lab established limits.)
 - Samples (≤ 10)
 - CCV ($\leq \pm 15\%$)
 - Samples (≤ 7)
 - MD (Compare results with lab established limits.)
 - MS (Compare results with lab established limits.)
 - MSD (Compare results with lab established limits.)
 - (2) Middle Batch: (≤ 20 samples of similar matrix.)
 - CCV ($\leq \pm 15\%$)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Table 3 or manufacturer/lab established limits.)
 - Samples (≤ 10)
 - CCV ($\leq \pm 15\%$)
 - Samples (≤ 7)
 - MD (Compare results with lab established limits.)
 - MS (Compare results with lab established limits.)
 - MSD (Compare results with lab established limits.)
 - (3) Final Batch: (≤ 20 samples of similar matrix.)
 - BFB
 - CCV ($\leq \pm 15\%$)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Table 3 or manufacturer/lab established limits.)
 - Samples (≤ 10)
 - CCV ($\leq \pm 15\%$)
 - Samples (≤ 7)
 - MD (Compare results with lab established limits.)
 - MS (Compare results with lab established limits.)
 - MSD (Compare results with lab established limits.)
 - CCV ($\leq \pm 15\%$)
9. Other Criteria:
 - (1) When doubt exists in compound identification, second column or GC/MS confirmation should be used.
 - (2) Establish retention time windows at $\pm 3\sigma$ with three injections throughout 72 hours.
 - (3) Establish %R for surrogates, LCS, BS, and MS, and RPD for BD, MD, and MSD.

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CHART J-5.2 Aromatic Volatile Organic Compounds by GC (8020)

1. Number of Analytes: 8
2. Preservation/Storage Conditions: pH<2 with HCl, H₂SO₄, or NaHSO₄ (aqueous). Na₂S₂O₃ if chlorine present (aqueous); stored at 4°C.
3. Holding Time: 14 days.
4. Amount for Extraction: 5 mL (aqueous) and 5 grams (solid) by 5030A.
5. Method of Validation:
 - (1) Extract and analyze four replicates of QC check standard. Compare results with Table 3.
 - (2) MDL (Table 1) shall be empirically established and verified semiannually for each matrix.
 - (3) Linear calibration range shall be established and verified semiannually.
6. Standards:
 - (1) Standard Solution Expiration: Stock standards: six months; calibration standards: 24 hours if no headspace
 - (2) Internal Standards: Optional. If used, α,α,α -trifluorotoluene is recommended.
 - (3) Surrogate Standards: Add surrogates (bromochlorobenzene, bromofluorobenzene, fluorobenzene, difluorobenzene, and α,α,α -trifluorotoluene are recommended) to encompass range of temperature program. Results within lab established control limits.
 - (4) QC Check Standards: If MS/MSD results fall outside control limits, a QC check standard must be analyzed and fall within those ranges designated in Table 3.
7. Calibration:
 - (1) Initial Calibration: Minimum of five levels with the lowest near but above MDL; linear correlation coefficient ≥ 0.995 . If %RSD<20, linearity is assumed and average RF may be used.
 - (2) Continuing Calibration: Mid-level calibration standard run every ten samples and at the end of the analytical run. If not within $\pm 15\%$ of predicted response, recalibrate.
8. Analysis: An example of run log is listed below.
 - (1) Initial Batch: (≤ 20 samples of similar matrix.)
 - CCV ($\leq \pm 15\%$)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Table 3 or manufacturer/lab established limits.)
 - Samples (≤ 10)
 - CCV ($\leq \pm 15\%$)
 - Samples (≤ 7)
 - MD (Compare results with lab established limits.)
 - MS (Compare results with lab established limits.)
 - MSD (Compare results with lab established limits.)
 - (2) Middle Batch: (≤ 20 samples of similar matrix.)
 - CCV ($\leq \pm 15\%$)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Table 3 or manufacturer/lab established limits.)
 - Samples (≤ 10)
 - CCV ($\leq \pm 15\%$)
 - Samples (≤ 7)
 - MD (Compare results with lab established limits.)
 - MS (Compare results with lab established limits.)
 - MSD (Compare results with lab established limits.)
 - (3) Final Batch: (≤ 20 samples of similar matrix.)
 - CCV ($\leq \pm 15\%$)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Table 3 or manufacturer/lab established limits.)
 - Samples (≤ 10)
 - CCV ($\leq \pm 15\%$)
 - Samples (≤ 7)
 - MD (Compare results with lab established limits.)
 - MS (Compare results with lab established limits.)
 - MSD (Compare results with lab established limits.)
 - CCV ($\leq \pm 15\%$)
9. Other Criteria:
 - (1) When doubt exists in compound identification, second column or GC/MS confirmation should be used.
 - (2) Establish retention time windows at $\pm 3\sigma$ with three injections throughout 72 hours.
 - (3) Establish %R for surrogates, LCS, BS, and MS, and RPD for BD, MD, and MSD.

CHART J-5.3 Organochlorine Pesticides and PCBS by GC (8080)

1. Number of Analytes: 26
2. Preservation/Storage Conditions: Na₂S₂O₅ if chlorine present (aqueous); stored at 4°C.
3. Holding Time: Extraction: seven days (aqueous) and 14 days (solid). Analysis: 40 days after extraction.
4. Amount for Extraction: One liter (aqueous) by 3510A or 3520A. 30 grams (low level solid) or two grams (medium level solid) by 3540A or 3550.
5. Method of Validation:
 - (1) Extract and analyze four replicates of QC check standard. Compare results with Table 3.
 - (2) MDL (Table 1) shall be empirically established and verified semiannually for each matrix.
 - (3) Linear calibration range shall be established and verified semiannually.
6. Standards:
 - (1) Standard Solution Expiration: Stock standards: one year; calibration standards: six months.
 - (2) Internal Standards: Optional; no internal standards specified.
 - (3) Surrogate Standards: Two surrogates, decachlorobiphenyl (DCBP) and 2,4,5,6-tetrachloro-m-xylene (TCMX). Results must fall within laboratory established limits.
 - (4) QC Check Standards: If MS/MSD results fall outside control limits, a QC check standard must be analyzed and fall within those ranges designated in Table 3.
7. Calibration:
 - (1) Initial Calibration: Minimum of five levels with the lowest near but above MDL; linear correlation coefficient ≥ 0.995 . If %RSD <20 , linearity is assumed and average RF may be used.
 - (2) Continuing Calibration: Mid-level calibration standard run every ten samples and at the end of the analytical run. If not within $\pm 15\%$ of predicted response, recalibrate.
8. Analysis: An example of run log is listed below.
 - (1) Initial Batch: (≤ 20 samples of similar matrix.)
GC Column deactivation (GC not used for one day or more; primed at 20x mid-level standard.)
Instrument blank
DDT and Endrin degradation check standard (Breakdown $<20\%$.)
CCV ($\leq \pm 15\%$)
 - MB ($<MDL$, $<5\%$ of regulatory limits, or $<5\%$ of measured sample concentration.)
 - LCS (Table 3 or manufacturer/lab established limits.)
 - Samples (≤ 10)
 - CCV ($\leq \pm 15\%$)
 - Samples (≤ 7)
 - MD (Compare results with lab established limits.)
 - MS (Compare results with Tables ONE-39, 40 of SW-846, Rev. 0 or lab established limits.)
 - MSD (Compare results with Tables ONE-39, 40 of SW-846, Rev. 0 or lab established limits.)
 - (2) Middle Batch: (≤ 20 samples of similar matrix.)
 - CCV ($\leq \pm 15\%$)
 - MB ($<MDL$, $<5\%$ of regulatory limits, or $<5\%$ of measured sample concentration.)
 - LCS (Table 3 or manufacturer/lab established limits.)
 - Samples (≤ 10)
 - CCV ($\leq \pm 15\%$)
 - Samples (≤ 7)
 - MD (Compare results with lab established limits.)
 - MS (Compare results with Tables ONE-39, 40 of SW-846, Rev. 0 or lab established limits.)
 - MSD (Compare results with Tables ONE-39, 40 of SW-846, Rev. 0 or lab established limits.)
 - (3) Final Batch: (≤ 20 samples of similar matrix.)
 - CCV ($\leq \pm 15\%$)
 - MB ($<MDL$, $<5\%$ of regulatory limits, or $<5\%$ of measured sample concentration.)
 - LCS (Table 3 or manufacturer/lab established limits.)
 - Samples (≤ 10)
 - CCV ($\leq \pm 15\%$)
 - Samples (≤ 7)
 - MD (Compare results with lab established limits.)
 - MS (Compare results with Tables ONE-39, 40 of SW-846, Rev. 0 or lab established limits.)
 - MSD (Compare results with Tables ONE-39, 40 of SW-846, Rev. 0 or lab established limits.)
 - CCV ($\leq \pm 15\%$)
9. Other Criteria:
 - (1) Check for DDT and Endrin degradation. Breakdown must be $<20\%$ for packed column GC or $<15\%$ for capillary GC.
 - (2) Second column confirmation is required for all hits. If compound concentration in the extract >10 ng/mL, GC/MS confirmation could be used.
 - (3) Establish retention time windows at $\pm 3\sigma$ with three injections throughout 72 hours.
 - (4) Establish %R for surrogates, LCS, BS, and MS, and RPD for BD, MD, and MSD.

CHART J-5.4 Chlorinated Herbicides by GC (8150A)

1. Number of Analytes: 10
2. Preservation/Storage Conditions: Na₂S₂O₅ if chlorine present (aqueous); stored at 4°C.
3. Holding Time: Extraction: seven days (aqueous) and 14 days (solid). Analysis: 40 days after extraction.
4. Amount for Extraction: One liter (aqueous) and 50 grams (solid) by 8150A.
5. Method of Validation:
 - (1) Extract and analyze four replicates of QC check standard. Compare results with Table 3.
 - (2) MDL (Table 1) shall be empirically established and verified semiannually for each matrix.
 - (3) Linear calibration range shall be established and verified semiannually.
6. Standards:
 - (1) Standard Solution Expiration: Stock standards: one year; calibration standards: six months.
 - (2) Internal Standards: Optional; no internal standards specified.
 - (3) Surrogate Standards: One/two surrogates added to each sample (avoid use of deuterated analogs.) Results must fall within laboratory established limits.
 - (4) QC Check Standards: If MS/MSD results fall outside control limits, a QC check standard must be analyzed and fall within those ranges designated in Table 3.
7. Calibration:
 - (1) Initial Calibration: Minimum of five levels with the lowest near but above MDL; linear correlation coefficient ≥ 0.995 . If %RSD < 20, linearity is assumed and average RF may be used.
 - (2) Continuing Calibration: Mid-level calibration standard run every ten samples and at the end of the analytical run. If not within $\pm 15\%$ of predicted response, recalibrate.
8. Analysis: An example of run log is listed below.
 - (1) Initial Batch: (≤ 20 samples of similar matrix.)
 - CCV ($\leq \pm 15\%$)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Table 3 or manufacturer/lab established limits.)
 - Samples (≤ 10)
 - CCV ($\leq \pm 15\%$)
 - Samples (≤ 7)
 - MD (Compare results with lab established limits.)
 - MS (Compare results with lab established limits.)
 - MSD (Compare results with lab established limits.)
 - (2) Middle Batch: (≤ 20 samples of similar matrix.)
 - CCV ($\leq \pm 15\%$)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Table 3 or manufacturer/lab established limits.)
 - Samples (≤ 10)
 - CCV ($\leq \pm 15\%$)
 - Samples (≤ 7)
 - MD (Compare results with lab established limits.)
 - MS (Compare results with lab established limits.)
 - MSD (Compare results with lab established limits.)
 - (3) Final Batch: (≤ 20 samples of similar matrix.)
 - CCV ($\leq \pm 15\%$)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Table 3 or manufacturer/lab established limits.)
 - Samples (≤ 10)
 - CCV ($\leq \pm 15\%$)
 - Samples (≤ 7)
 - MD (Compare results with lab established limits.)
 - MS (Compare results with lab established limits.)
 - MSD (Compare results with lab established limits.)
 - CCV ($\leq \pm 15\%$)
9. Other Criteria:
 - (1) When doubt exists in compound identification, GC/MS or second column confirmation should be used.
 - (2) Establish retention time windows at $\pm 3\sigma$ with three injections throughout 72 hours.
 - (3) Establish %R for surrogates, LCS, BS, and MS, and RPD for BD, MD, and MSD.

CHART J-5.5 Volatile Organic Compounds by GC/MS (8240A)

1. Number of Analytes: 74 (Minimum: 35 in Table 2 of 8240, Rev. 0, 1986)
2. Preservation/Storage Conditions: pH<2 with HCl, H₂SO₄, or NaHSO₄ (aqueous). Na₂S₂O₅ if chlorine present (aqueous); stored at 4°C.
3. Holding Time: 14 days.
4. Amount for Extraction: 5 mL (aqueous) and 5 grams (solid) by 5030A.
5. Method of Validation:
 - (1) Extract and analyze four replicates of QC check standard. Compare results with Table 6.
 - (2) EQLs (Table 2) shall be empirically established and verified semiannually for each matrix.
 - (3) Linear calibration range shall be established and verified semiannually.
6. Standards:
 - (1) Standard Solution Expiration: Stock standards (except gases): six months; stock gas standards: two months; calibration standards: daily.
 - (2) Internal Standards: Bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d₅. RT must be within ±30 seconds from last calibration; area must be -50 to +100%.
 - (3) Surrogate Standards: 4-Bromofluorobenzene, 1,2-dichloroethane-d₄, and toluene-d₈. Recover limits in Table 8.
 - (4) QC Check Standards: If MS/MSD results fall outside control limits, a QC check standard must be analyzed and fall within those ranges designated in Table 6.
7. Calibration:
 - (1) GC/MS Tuning: 50 ng of 4-bromofluorobenzene (BFB) which meets the criteria given in Table 3.
 - (2) Initial Calibration: Minimum of five levels with the lowest near but above MDL. %RSD should be <30% for each CCC (1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethyl benzene, and vinyl chloride). RF>0.30 for SPCCs (chloromethane, 1,1-dichloroethane, chlorobenzene, and 1,1,2,2-tetrachloro-ethane) except 0.25 for bromoform.
 - (3) Continuing Calibration: Mid-level calibration standard run every 12 hours. RF>0.30 for SPCCs except 0.25 for bromoform. RF for each CCC must be <25% difference from initial calibration.
8. Analysis: An example of run log is listed below.
 - (1) Initial Batch: (≤20 samples of similar matrix.)
 - BFB tuning to meet criteria in Table 3
 - CCV (Mid-concentration calibration standard every 12 hours.)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Table 6 or manufacturer/lab established limits.)
 - Samples
 - BFB (Table 3; every 12 hours.)
 - CCV (Mid-concentration calibration standard every 12 hours.)
 - MD (Compare results with lab established limits.)
 - MS (Compare results with Tables ONE-39, 40 of SW-846, Rev. 0 or lab established limits.)
 - MSD (Compare results with Tables ONE-39, 40 of SW-846, Rev. 0 or lab established limits.)
 - (2) Middle or Final Batch: (≤20 samples of similar matrix.)
 - BFB (Table 3; every 12 hours.)
 - CCV (Mid-concentration calibration standard every 12 hours.)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Table 6 or manufacturer/lab established limits.)
 - Samples
 - BFB (Table 3; every 12 hours.)
 - CCV (Mid-concentration calibration standard every 12 hours.)
 - MD (Compare results with lab established limits.)
 - MS (Compare results with Tables ONE-39, 40 of SW-846, Rev. 0 or lab established limits.)
 - MSD (Compare results with Tables ONE-39, 40 of SW-846, Rev. 0 or lab established limits.)
9. Other Criteria:
 - (1) Compound ID: All ions >10% intensity must be ±20% of standard; ±0.06 RRT units of standard RRT.
 - (2) Establish %R for surrogates, LCS, BS, and MS, and RPD for BD, MD, and MSD.
 - (3) The most recent version of the EPA/NIST Mass Spectral Library or equivalent should be available.

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CHART J-5.6 Semivolatile Organic Compounds by GC/MS (8270A)

1. Number of Analytes: 233 (Minimum: 65 in Table 2, 8270, Rev. 0, 1986.)
2. Preservation/Storage Conditions: Na₂S₂O₃ if chlorine present (aqueous); stored at 4°C.
3. Holding Time: Extraction: seven days (aqueous) and 14 days (solid). Analysis: 40 days after extraction.
4. Amount for Extraction: One liter (aqueous) by 3510A or 3520A at pH>11 and pH<2. 30 grams (low level solid) or two grams (medium level solid) by 3540A or 3550.
5. Method of Validation:
 - (1) Extract and analyze four replicates of QC check standard. Compare results with Table 6.
 - (2) EQLs (Table 2) shall be empirically established and verified semiannually for each matrix.
 - (3) Linear calibration range shall be established and verified semiannually.
6. Standards:
 - (1) Standard Solution Expiration: Stock standards: one year; calibration standards: one year; daily continuing calibration standards: one week.
 - (2) Internal Standards: 1,4-Dichlorobenzene-d₄, naphthalene-d₈, acenaphthene-d₁₀, crysene-d₁₂, and perylene-d₁₂. RT must be within ±30 seconds from last calibration; area must be -50 to
 - (3) Surrogate Standards: Nitrobenzene-d₅, 2-fluorobiphenyl, p-terphenyl-d₁₄, phenol-d₅, 2-fluorophenol, and 2,4,6-tribromophenol. Recover limits in Table 8.
 - (4) QC Check Standards: If MS/MSD results fall outside control limits, a QC check standard must be analyzed and fall within those ranges designated in Table 6.
7. Calibration:
 - (1) GC/MS Tuning: 50 ng of decafluorotriphenyl phosphine (DFTPP) which meets the criteria given in Table 3. The standard should also contain 4,4'-DDT, pentachlorophenol, and benidine to verify injection port inertness and GC column performance. (Degradation of DDT <20%. No peak tailing.)
 - (2) Initial Calibration: Minimum of five levels with the lowest near but above MDL. %RSD should be <30% for each compound and must be <30% for each CCC (Table 4). Retention time for each compound agrees within 0.06 relative retention time unit. RF>0.05 for SPCCs (N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitro-phenol, and 4-nitrophenol.)
 - (3) Continuing Calibration: Mid-level calibration standard run every 12 hours. RF>0.05 for SPCCs. RF for each CCC must be <30% difference from initial calibration.
8. Analysis: An example of run log is listed below.
 - (1) Initial Batch: (≤ 20 samples of similar matrix.)
 - DFTPP tuning to meet-criteria in Table 3
 - CCV (Mid-concentration calibration standard.)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Table 6 or manufacturer/lab established limits.)
 - Samples
 - DFTPP (Table 3; every 12 hours.)
 - CCV (Mid-concentration calibration standard every 12 hours.)
 - MD (Compare results with lab established limits.)
 - MS (Compare results with Tables ONE-39, 40 of SW-846, Rev. 0 or lab established limits.)
 - MSD (Compare results with Tables ONE-39, 40 of SW-846, Rev. 0 or lab established limits.)
 - (2) Middle or Final Batch: (≤ 20 samples of similar matrix.)
 - DFTPP (Table 3; every 12 hours.)
 - CCV (Mid-concentration calibration standard every 12 hours.)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Table 6 or manufacturer/lab established limits.)
 - Samples
 - DFTPP (Table 3; every 12 hours.)
 - CCV (Mid-concentration calibration standard every 12 hours.)
 - MD (Compare results with lab established limits.)
 - MS (Compare results with Tables ONE-39, 40 of SW-846, Rev. 0 or lab established limits.)
 - MSD (Compare results with Tables ONE-39, 40 of SW-846, Rev. 0 or lab established limits.)
9. Other Criteria:
 - (1) Compound ID: All ions >10% intensity must be ±20% of standard; ±0.06 RRT units of standard RRT.
 - (2) Establish %R for surrogates, LCS, BS, and MS, and RPD for BD, MD, and MSD.
 - (3) The most recent Version of the EPA/NIST Mass Spectral Library or equivalent should be available.

CHART J-5.7 Metals by ICP (6010A)

1. Number of Analytes: 26 metals
2. Preservation/Storage Conditions: pH<2 with HNO₃; stored at 4°C (solid).
3. Holding Time: Six months.
4. Amount for Digestion: 100 mL (aqueous) by 3005A (aqueous total recoverable or dissolved metals), 3010A (aqueous total metals), 3040 (dissolution procedures); and 1.00-2.00 grams (solid) by 3050A (solid total metals).
5. Method of Validation: IDL (listed in Table 1 of Method 6010A) shall be empirically established and verified for each matrix.
6. Standards:
 - (1) Standard Solution Expiration: Stock standards: specified by manufacturer; must be monitored weekly; calibration standards: prepare fresh at time of use.
7. Calibration:
 - (1) Initial Calibration: Per instrument manufacturer's specifications (should consist of a daily minimum of three levels plus a calibration blank.) Before beginning the sample run, reanalyze the highest mixed calibration standard. Concentration values should be $\leq \pm 5\%$ of the true values or the established control limits, whichever is lower.
 - (2) Continuing Calibration: A mid-level, second source CCV run every ten samples and at the end of the analytical run; %R=90-110.
 - (3) Interference check solution (ICS): Used to spike sample with the element of interest at concentrations of 10x IDL. Run at the beginning and the end of an analytical run or twice during every 8-hour work shift, whichever is more frequent.
8. Analysis: An example of run log is listed below.
 - (1) Initial Batch: (≤ 20 samples of similar matrix.)
 - Minimum of three level calibration plus a calibration blank
 - Highest mixed standard ($\leq \pm 5\%$ of true value)
 - ICS ($\leq \pm 20\%$ of true value)
 - LCS (Based on control chart or $\leq \pm 20\%$ prior to establishment of control chart.)
 - Samples (≤ 10)
 - Calibration blank ($< 3\sigma$ of the mean blank value.)
 - CCV ($\leq \pm 10\%$)
 - Samples (≤ 6)
 - MB ($< \text{MDL}$, $< 5\%$ of regulatory limits, or $< 5\%$ of measured sample concentration.)
 - MD (RPD < 20)
 - MS (%R=80-120)
 - MSD (RPD < 20 , %R=80-120.)
 - (2) Middle Batch: (≤ 20 samples of similar matrix.)
 - Calibration blank ($< 3\sigma$ of the mean blank value.)
 - CCV ($\leq \pm 10\%$)
 - LCS (Based on control chart or $\leq \pm 20\%$ prior to establishment of control chart.)
 - Samples (≤ 10)
 - Calibration blank ($< 3\sigma$ of the mean blank value.)
 - CCV ($\leq \pm 10\%$)
 - Samples (≤ 6)
 - MB ($< \text{MDL}$, $< 5\%$ of regulatory limits, or $< 5\%$ of measured sample concentration.)
 - MD (RPD < 20)
 - MS (%R=80-120)
 - MSD (RPD < 20 , %R=80-120.)
 - (3) Final Batch: (20 samples of similar matrix.)
 - Calibration blank ($< 3\sigma$ of the mean blank value.)
 - CCV ($\leq \pm 10\%$)
 - LCS (Based on control chart or $\leq \pm 20\%$ prior to establishment of control chart.)
 - Samples (≤ 10)
 - Calibration blank ($< 3\sigma$ of the mean blank value.)
 - CCV ($\leq \pm 10\%$)
 - ICS (Additional ICS, if more than eight hours.)
 - Samples (≤ 6)
 - MB ($< \text{MDL}$, $< 5\%$ of regulatory limits, or $< 5\%$ of measured sample concentration.)
 - MD (RPD < 20)
 - MS (%R=80-120)
 - MSD (RPD < 20 , %R=80-120)
 - Calibration blank ($< 3\sigma$ of the mean blank value.)
 - ICS ($\leq \pm 20\%$)
 - CCV ($\leq \pm 10\%$)

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CHART J-5.7 Metals by ICP (6010A) (continued)

9. Other Criteria:

- (1) Test for matrix interference with each matrix using a 5-fold serial dilution test (if >50x IDL), Percent difference $\leq \pm 10\%$; or a (10-100x IDL) post-digestion spike test, %R=75-125.
- (2) Use MSA to compensate for matrix interferences.
- (3) Use multiple exposures for both calibration and sample analysis.
- (4) Establish %R for LCS, BS, and MS, and RPD for BD, MD, and MSD.

CHART J-5.8 Metals by Flame and Graphite Furnace AA (7000s)

1. Number of Analytes: 27 metals
2. Preservation/Storage Conditions: pH<2 with HNO₃; stored at 4°C (solid).
3. Holding Time: Six months.
4. Amount for Digestion: 100 mL (aqueous) by 3005A (aqueous total recoverable metals or dissolved metals by FLAA), 3010A (aqueous total metals by FLAA), 3020 (aqueous total metals by GFAA, except As by 7060 and Se by 7740), 3040 (dissolution procedures for AA); and 1.00-2.00 grams (solid) by 3050A (solid total metals by FLAA and GFAA).
5. Method of Validation: MDL (listed in Table 1 of Method 7000A) shall be empirically established and verified semiannually for each matrix.
6. Standards:
 - (1) Standard Solution Expiration: Stock standards: specified by manufacturer; must be monitored weekly; calibration standards: prepare fresh at time of use.
7. Calibration:
 - (1) Initial Calibration: Minimum of a daily three level calibration plus a calibration blank. Verify with a calibration blank and a mid-level ICV from a second source; %R=90-110.
 - (2) Continuing Calibration: A mid-level, second source CCV or QC check standard run every ten samples and at the end of the analytical run; %R=80-120.
8. Analysis: An example of run log is listed below.
 - (1) Initial Batch: (≤20 samples of similar matrix.)
 - Minimum of a three level calibration plus a calibration blank
 - Calibration blank (<IDL)
 - ICV (<±10%)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Based on control chart or <±20% prior to establishment of control chart.)
 - Samples (≤10)
 - CCV (<±20%)
 - Samples (≤7)
 - MD (RPD<20)
 - MS (%R=75-125)
 - MSD (RPD<20, %R=75-125.)
 - (2) Middle Batch: (≤20 samples of similar matrix.)
 - CCV (<±20%)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Based on control chart or <±20% prior to establishment of control chart.)
 - Samples (≤10)
 - CCV (<±20%)
 - Samples (≤7)
 - MD (RPD<20)
 - MS (%R=75-125)
 - MSD (RPD<20, %R=75-125.)
 - (3) Final Batch: (≤20 samples of similar matrix.)
 - CCV (<±20%)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Based on control chart or <±20% prior to establishment of control chart.)
 - Samples (≤10)
 - CCV (<±20%)
 - Samples (≤7)
 - MD (RPD<20)
 - MS (%R=75-125)
 - MSD (RPD<20, %R=75-125.)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - CCV (<±20%)
9. Other Criteria:
 - (1) Test for matrix interference with each batch using a 5-fold (1+4) dilution test (if sample >25x MDL); percent difference <10%. If dilution test fails or all samples in the batch <10x MDL, perform a (2-5x sample or 20x MDL) spike recovery test; %R=85-115.
 - (2) Use MSA to compensate for multiplicative interferences, i.e., matrix or physical interferences.
 - (3) Use Zeeman background correction for additive interferences, i.e., nonspecific absorption and scattering.
 - (4) Establish %R for LCS, BS, and MS, and RPD for BD, MD, and MSD.

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CHART J-5.9 Mercury by Cold Vapor AA (7470/7471)

1. Number of Analytes: Mercury (Hg)
2. Preservation/Storage Conditions: pH<2 with HNO₃; stored at 4°C (solid).
3. Holding Time: 28 days.
4. Amount for Digestion: 100 mL (aqueous) and 0.2 grams (solid) by 7470 (aqueous) and 7471 (solid).
5. Method of Validation: MDL (0.0002 mg/L listed in Section 1.0) shall be empirically established and verified semiannually for each matrix.
6. Standards:
 - (1) Standard Solution Expiration: Stock standards: specified by manufacturer; must be monitored weekly; calibration standards: prepare fresh at time of use.
7. Calibration:
 - (1) Initial Calibration: Minimum of a daily five level calibration plus a calibration blank. Verify with a calibration blank and a mid-level ICV from a second source: %R=90-110.
 - (2) Continuing Calibration: A mid-level, second source CCV or QC check standard run every ten samples and at the end of the analytical run; %R=80-120.
8. Analysis: An example of run log is listed below.
 - (1) Initial Batch: (≤20 samples of similar matrix.)
 - Minimum of a five level calibration plus a calibration blank
 - Calibration blank (<IDL)
 - ICV (<±10%)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Based on control chart or <±20% prior to establishment of control chart.)
 - Samples (≤10)
 - CCV (<±20%)
 - Samples (≤7)
 - MD (RPD<20)
 - MS (%R=75-125)
 - MSD (RPD<20, %R=75-125.)
 - (2) Middle Batch: (≤20 samples of similar matrix.)
 - CCV (<±20%)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Based on control chart or <±20% prior to establishment of control chart.)
 - Samples (≤10)
 - CCV (<±20%)
 - Samples (≤7)
 - MD (RPD<20)
 - MS (%R=75-125)
 - MSD (RPD<20, %R=75-125.)
 - (3) Final Batch: (≤20 samples of similar matrix.)
 - CCV (<±20%)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Based on control chart or <±20% prior to establishment of control chart.)
 - Samples (≤10)
 - CCV (<±20%)
 - Samples (≤7)
 - MD (RPD<20)
 - MS (%R=75-125)
 - MSD (RPD<20, %R=75-125.)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - CCV (<±20%)
9. Other Criteria:
 - (1) Test for matrix interference with each batch using a 5-fold (1+4) dilution test (if >25x MDL): percent difference <10%. If dilution test fails or all samples in the batch <10x MDL, perform a (2-5x sample or 20x MDL) spike recovery test; %R=85-115.
 - (2) Use MSA to compensate for matrix interferences.
 - (3) Establish %R for LCS, BS, and MS, and RPD for BD, MD, and MSD.

CHART J-5.10 Total and Amenable Cyanide by Calorimetry (9010A)

1. Number of Analytes: Total CN and CN amenable to chlorination
2. Preservation/Storage Conditions: pH ≥ 12 with NaOH. NaAsO₂ or ascorbic acid if oxidizing agents present; stored at 4°C.
3. Holding Time: 14 days.
4. Amount for Preparation: 500 mL (1,000 mL if both total and amenable CN) (aqueous) and 1-5 grams (2-10 grams if both total and amenable CN) (solid) by distillation procedures in 9010.
5. Method of Validation: MDL (0.02 mg/L listed in Section 1.0) shall be empirically established and verified semiannually for each matrix.
6. Standards: Stock standards expiration: not specified; calibration standards expiration: daily.
7. Calibration:
 - (1) Samples contain no sulfides:
 - (a) Initial Calibration: Daily minimum of six levels and a calibration blank, plus a minimum of two of the above standards (high and low) distilled. The distilled ones should be $\leq \pm 10\%$ of undistilled ones.
 - (b) Continuing Calibration: Mid-level calibration standard run every batch and should be $\leq \pm 15\%$ of expected value.
 - (2) Samples contain sulfides:
 - (a) Initial calibration: Daily six standards and calibration blank. All standards are distilled as the samples using the method of standard additions.
 - (b) Continuing Calibration: Mid-level calibration standard run every batch and should be $\leq \pm 15\%$ of expected value.
8. Analysis: An example of run log is listed below.
 - (1) Samples contain no sulfides:
 - (a) Initial Batch: (≤ 20 samples of similar matrix.)
 - Minimum of six level plus blank calibration
 - Check standards (Second source, middle level, no distillation, $\leq \pm 15\%$.)
 - MB (Distilled; $< \text{MDL}$, $< 5\%$ of regulatory limits, or $< 5\%$ of measured sample concentration.)
 - LCS (Distilled; compare results with manufacturer/lab established limits.)
 - Samples (Distilled.)
 - MS (Distilled; compare results with lab established limits.)
 - MD (Distilled, $\leq \pm 20\%$)
 - (b) Middle Batch: (≤ 20 samples of similar matrix.)
 - Check standards (Second source, middle level, no distillation, $\leq \pm 15\%$.)
 - MB (Distilled; $< \text{MDL}$, $< 5\%$ of regulatory limits, or $< 5\%$ of measured sample concentration.)
 - LCS (distilled; compare results with manufacturer/lab established limits.)
 - Samples (Distilled.)
 - MS (Distilled; compare results with lab established limits.)
 - MD (Distilled, $\leq \pm 20\%$)
 - (c) Final Batch: (≤ 20 samples of similar matrix.)
 - Check standards (Second source, middle level, no distillation, $\leq \pm 15\%$.)
 - MB (Distilled; $< \text{MDL}$, $< 5\%$ of regulatory limits, or $< 5\%$ of measured sample concentration.)
 - LCS (Distilled; compare results with manufacturer/lab established limits.)
 - Samples (Distilled.)
 - MS (Distilled; compare results with lab established limits.)
 - MD (Distilled, $\leq \pm 20\%$)
 - Check standards (Second source, middle level, no distillation, $\leq \pm 15\%$.)
 - (2) Samples contain sulfides:
 - (a) Initial Batch: (≤ 20 samples of similar matrix.)
 - Minimum of six level plus blank calibration using MSD
 - Check standards (Second source, middle level, no distillation, $\leq \pm 15\%$.)
 - MB (Distilled; $< \text{MDL}$, $< 5\%$ of regulatory limits, or $< 5\%$ of measured sample concentration.)
 - LCS (Distilled; compare results with manufacturer/lab established limits.)
 - Samples (Distilled.)
 - MS (Distilled; compare results with lab established limits.)
 - MD (Distilled, $\leq \pm 20\%$)
 - (b) Middle Batch: (≤ 20 samples of similar matrix.)
 - Check standards (Second source, middle level, no distillation, $\leq \pm 15\%$.)
 - MB (Distilled; $< \text{MDL}$, $< 5\%$ of regulatory limits, or $< 5\%$ of measured sample concentration.)
 - LCS (Distilled; compare results with manufacturer/lab established limits.)
 - Samples (Distilled.)
 - MS (Distilled; compare results with lab established limits.)
 - MD (Distilled, $\leq \pm 20\%$)

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CHART J-5.10 Total and Amenable Cyanide by Calorimetry (9010A)
(continued)

- (c) Final Batch: (≤20 samples of similar matrix.)
 - Check standards (Second source, middle level, no distillation, $<\pm 15\%$.)
 - MB (Distilled; $<MDL$, $<5\%$ of regulatory limits, or $<5\%$ of measured sample concentration.)
 - LCS (Distilled; compare results with manufacturer/lab established limits.)
 - Samples (Distilled.)
 - MS (Distilled; compare results with lab established limits.)
 - MD (Distilled, $<\pm 20\%$)
 - Check standards (Second source, middle level, no distillation, $<\pm 15\%$.)
- 9. Other Criteria:
 - (1) Use MSA to compensate for matrix interferences.
 - (2) Establish %R for check standards, LCS, BS, and MS, and RPD for BD and MD.

**CHART J-5.11 Total Organic Carbon by a Carbonaceous Analyzer
(9060)**

1. Number of Analytes: No specific compounds.
2. Preservation/Storage Conditions: pH<2 with HCL or H₂SO₄. Protect from light and atmospheric O₂; stored at 4°C.
3. Holding Time: 28 days.
4. Amount for Extraction: 50 mL.
5. Method of Validation: MDL (1 mg/L listed in Section 1.0) shall be empirically established and verified semiannually for each matrix.
6. Standards: Standard Solution Expiration: Not specified.
7. Calibration:
 - (1) Initial Calibration: Per instrument manufacturer's specifications, correlation coefficient ≥ 0.995 .
 - (2) Continuing calibration: Percent difference <10% of initial calibration.
8. Analysis: An example of run log is listed below.
 - (1) Initial Batch: (≤20 samples of similar matrix.)
 - Initial calibration
 - CCV (Mid-level; independently prepared; percent difference <10% of initial calibration.)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Based on lab established control limits and should be <10% difference.)
 - Samples
 - MS (Based on lab established control limits.)
 - MD (Based on lab established control limits.)
 - (2) Middle Batch: (≤20 samples of similar matrix.)
 - CCV (Mid-level; independently prepared; percent difference <10% of initial calibration.)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Based on lab established control limits and should be <10% difference.)
 - Samples
 - MS (Based on lab established control limits.)
 - MD (Based on lab established control limits.)
 - (3) Final Batch: (≤20 samples of similar matrix.)
 - CCV (Mid-level; independently prepared; percent difference <10% of initial calibration.)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Based on lab established control limits and should be <10% difference.)
 - Samples
 - MS (Based on lab established control limits.)
 - MD (Based on lab established control limits.)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - CCV (Mid-level; independently prepared; percent difference <10% of initial calibration.)
9. Other Criteria: Establish %R for LCS, BS, and MS, and RPD for BD and MD.

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**CHART J-5.12 Total Recoverable Petroleum Hydrocarbons by IR
(418.1)**

1. Number of Analytes: Non-polar petroleum hydrocarbons. No specific compounds.
2. Preservation/Storage Conditions: pH<2 with HCL (aqueous); stored at 4°C.
3. Holding Time: 28 days.
4. Amount for Extraction: 1,000 mL (aqueous) by 418.1 and 20 grams (solid) by 9071 steps 7.1 thru 7.11 (Soxhlet extraction, 3540A.)
5. Method of Validation: MDL (1 mg/L listed in Section 1.0) shall be empirically established and verified annually for each matrix.
6. Standards:
 - (1) Reference oil: Mixture of 15.0 mL n-hexadecane, 15.0 mL isooctane, and 10.0 mL chlorobenzene.
 - (2) Standard Solution Expiration: Stock Standards: six months; working standards: one week.
7. Calibration:
 - (1) Initial Calibration: Daily minimum of four levels plus a calibration blank; correlation coefficient >0.995.
 - (2) Continuing calibration: Percent difference <10% of initial calibration.
8. Analysis: An example of run log is listed below.
 - (1) Initial Batch: (≤20 samples of similar matrix.)
 - Initial calibration
 - CCV (Mid-level; independently prepared; percent difference <10% of initial calibration.)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Based on lab established control limits and should be <10% difference.)
 - Samples
 - MS (Based on lab established limits.)
 - MD (Based on lab established limits.)
 - (2) Middle Batch: (≤20 samples of similar matrix.)
 - CCV (Mid-level; independently prepared; percent difference <10% of initial calibration.)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Based on lab established control limits and should be <10% difference.)
 - Samples
 - MS (Based on lab established limits.)
 - MD (Based on lab established limits.)
 - (3) Final Batch: (≤20 samples of similar matrix.)
 - CCV (Mid-level; independently prepared; percent difference <10% of initial calibration.)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - LCS (Based on lab established control limits and should be <10% difference.)
 - Samples
 - MS (Based on lab established limits.)
 - MD (Based on lab established limits.)
 - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
 - CCV (Mid-level; independently prepared; percent difference <10% of initial calibration.)
9. Other Criteria:
 - (1) Acidify solid samples to pH=2 with HCL.
 - (2) Use MgSO₄•H₂O for solid samples.
 - (3) Establish %R for LCS, BS, and MS, and RPD for BD and MD.

CHART J-6 Data Reduction, Validation, and Reporting

1. SOPs
2. Computerized Data Reduction (Manually checked.)
3. Multiple Levels of Data Review
 - a. Analyst or peer (100%)
 - b. Supervisor ($\geq 10\%$)
 - c. QA Officer ($\geq 10\%$)
 - d. Lab Manager/Director ($\geq 10\%$)
4. Data Qualifier Flags
5. Report Generation and Archives
 - a. Prenumbered, permanently bound notebooks.
 - b. Corrections do not obliterate original data.
 - c. Revised entry is signed or initialed and dated.
 - d. Records are traceable, retrievable, legible, and complete.
 - e. All data and reports stored in a secured area for a minimum of three years after final reports.
6. Corrective Actions and Documentation

CHART J-7 Performance and System Audits

1. SOPs
2. Designated Internal Auditor
3. Performance Audit
 - a. External QA
 - b. Internal QA
 - (1) Initial evaluation of new analysts
 - (2) Periodical audit of experienced analysts
 - (3) Single blind PE samples
 - (4) Double blind PE samples
 - c. Round robin testing
 - d. Corrective actions
 - e. Documentation
4. System Audit
 - a. Methodologies
 - (1) New method
 - (2) Modified method
 - (3) New instrument
 - b. Documentation
5. Control Charts: Established for each type of QC parameters, methodologies, and matrices; updated quarterly or when 20 new data points are obtained.
 - a. MB
 - b. LCS
 - c. MD
 - d. MS
 - e. MSD
 - f. Surrogate
 - g. Others

CHART J-8 Laboratory Safety

1. Safety and Chemical Hygiene Plan
 - a. Safety meeting
 - b. Safety inspection
 - c. Fire drill
2. Safety Equipment
 - a. Eyewash fountain
 - b. Emergency shower
 - c. Safety glasses and gloves
 - d. Fire alarm
 - e. Fire Extinguisher
 - f. Emergency light
 - g. Flammable material storage
 - h. Hazardous area escape
 - i. OSHA signs
 - j. First aid kit

CHART J-9 Waste Management

1. SOPs
 - a. Waste stream analysis
 - b. Waste segregation program
 - c. Waste recycle program
2. Are residual USACE samples properly disposed of?
3. Does the lab have a Hazardous Waste Coordinator? (Federal RCRA Compliance Checklist, Appendix E, Section 7.)
4. Is the lab a conditionally exempted small quantity generator?
 - a. The lab generates less than 100 kg per month of hazardous waste or less than 1 kg per month of acute hazardous waste.
 - b. There is never more than 1,000 kg stored on site.
 - c. Waste is sent to a TSDF, a facility that beneficially reuse the waste, or a state permitted facility.
5. Are there records to substantiate the above claims?
6. Does the lab use a manifest when shipping hazardous waste?
7. Is aqueous waste disposed of into a sanitary sewer only if it is neutralized and approved in writing by the sewer authority?
8. Does the lab have the following documents for review?
 - a. USEPA Notification Form 8700-12
 - b. USEPA Identification Number
 - c. Small Quantity Generator Permit
 - d. RCRA Part A Permit
 - e. RCRA Part B permit
 - f. NPDES Permit
 - g. Manifests
 - h. Waste Analysis Records
 - i. Land Ban Records
 - j. Exception Reports
 - k. Biennial Reports
 - l. Annual Reports
 - m. Training and Personnel Files
 - n. Contingency Plan/SPCC Plan
 - o. Agreements with Local Emergency Authorities
 - p. Used Oil Records
 - q. Hazardous Waste Management Plan

CHART J-10 Government QA Functions (Applicable to government QA labs only.)

1. Project Coordination
 - a. Designated coordinator
 - b. Review CDAPS
 - c. DQO clarification
2. QA Activities
 - a. Review/comment on project documents
 - b. Attend project meeting
 - c. Perform site visits
 - d. Receive/review government QA samples
 - e. Analyze government QA samples
 - f. Evaluate contractor QC data
 - g. Prepare CQARS
 - h. Other activities
3. CQAR Preparation
 - a. Evaluation Parameters
 - (1) Precision (RPD based on MD, MSD, and BSD/LCSD if not enough samples.)
 - (2) Accuracy (Spike recovery based on LCS, MS/MSD, surrogates, and BS/BSD if not enough samples.)
 - (3) Representativeness (Holding time, MB, and MQ/MSD.)
 - (4) Comparability (Analytical method, MDL, precision, accuracy, and reporting unit.)
 - (5) Completeness (COC, holding times, MDL, MB, LCS, MS, MD/MSD, and surrogates.)
 - (6) Others
 - b. Evaluation Criteria
 - c. Agreement between contractor and government data
 - d. Timely release (Within 20 working days after receipt of contractor's final QC data, but before the completion of contractor's final engineering report.)
4. Contract Management (Appendix L)
 - a. Prepare SOW for new contract
 - b. Evaluate and select contractor
 - c. Assess contractor's data quality
 - d. Request corrective actions
 - e. Suspend/terminate contract